



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : C03C 25/02	A1	(11) International Publication Number: WO 00/00446 (43) International Publication Date: 6 January 2000 (06.01.00)
(21) International Application Number: PCT/NL99/00383 (22) International Filing Date: 21 June 1999 (21.06.99) (30) Priority Data: 1009503 26 June 1998 (26.06.98) NL (71) Applicant (for all designated States except US): PLASMA OPTICAL FIBRE B.V. [NL/NL]; Zwaanstraat 1, NL-5651 CA Eindhoven (NL). (72) Inventors; and (75) Inventors/Applicants (for US only): BREULS, Antonius, Henricus, Elisabeth [NL/NL]; Mauritslaan 6, NL-6129 EM Urmond (NL). DE FOUW, Marinus, Jacob [NL/NL]; Gotthardpas 34, NL-5624 NE Eindhoven (NL). (74) Agents: VAN KAN, J., J., H. et al.; Algemeen Octrooibureau, World Trade Center, Pastoor Petersstraat 160, NL-5612 LV Eindhoven (NL).		(81) Designated States: AE, AL, AM, AT, AT (Utility model), AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, CZ (Utility model), DE, DE (Utility model), DK, DK (Utility model), EE, EE (Utility model), ES, FI, FI (Utility model), GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SK (Utility model), SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: A METHOD OF APPLYING A PROTECTIVE ORGANIC COATING TO AN OPTICAL GLASS FIBRE (57) Abstract <p>The invention relates to a method of applying a protective organic coating to an optical glass fibre, said glass fibre is drawn from a preform and passed through a liquid which contains the material for forming said organic coating, once the amount of liquid coating material to be applied to the fibre has been adjusted, said coating material is hardened, while a gas is passed along the liquid wherein nitrous oxide (an N₂O-containing gas) is used as said gas. The invention also relates to the coated optical glass fibre, produced by that method.</p>		

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Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
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A method of applying a protective organic coating to an optical glass fibre.

The invention relates to a method of applying a protective organic coating to an optical glass fibre or to a coated optical glass fibre, wherein said glass fibre is drawn from a preform and passed through a liquid which contains the material for forming said organic coating, once the amount of liquid coating material to be applied to the fibre has been adjusted, said coating material is hardened and a gas is passed along the coating material.

A method of this kind is known from EP-A-0 261 772. In the claims of said patent application it is stated that CO₂ is used as said gas, thus minimizing the number of air inclusions that may form upon forming of the coating. The surface of the coating material of the glass fibre is conditioned by means of CO₂. It is stated in the introduction of EP-A-0 261 772 that various gases may be used, such as nitrogen, carbon dioxide, noble gases, especially xenon, neon and argon, and chemically inert gaseous hydrocarbons such as chloroform, Freon (brand name), halogen hydrocarbons or other chlorine- or fluorine-substituted hydrocarbons. In particular, however, CO₂ is used.

A method of the above kind is also known from EP-B-0 200 256, wherein it is indicated that xenon and dichlorodifluoromethane are gases which are usable within this framework.

Jochem et al "High-speed bubble-free coating of optical fibres on a short drawing tower" (IOOC and ECOC), Venice, 1985, part 1, pages 515-518, Istituto Internazionale Delle Comunicazioni discloses the use of several gases in such a process to draw optical fibres. In table 2 one mentioned air, He, A, Xe and CCl₂F₂. However Jochem et al did not indicate the gascomposition used according to the invention.

From EP-A-0 635 554 it is known that an optical fibre can be coated with hydrogen silsesquioxane by heating the fiber at a temperature of 50-1000 °C during up to 6 hours. The heating may be conducted at any pressure and under oxidizing or non-oxidizing gaseous environment usch as air, O₂, an inert gas (N₂, etc.), ammonia, amines, moisture, N₂O, hydrogen and hydrocarbons. From this disclosure the specific gas used according to the invention can not be derived.

Further research has shown that a higher-quality bond of the organic material to the glass fibre is obtained by using a specific gas. According to the invention, the method as stated in the introduction is therefore characterized in that nitrous oxide (an N_2O -containing gas) is used as said gas. The term nitrous oxide as used herein should be understood to mean a gas which contains at least 50% N_2O .

Preferably, the gas is introduced at the upper side of the device for applying the organic coating material to the fibre and carried downstream along with the glass fibre. We also found that with the method according to the invention it also is possible to apply a second or third organic coating layer to an already coated fibre. The amount of gas supplied to the liquid organic coating material depends on the construction of the device for applying the coating material and the drawing speed. Nevertheless this amount must be sufficient for preventing entrained air, that comes along with the fibre, to become entrapped in the coating. This amount of gas can be minimized by using specific nozzles or a small diameter shaft.

The invention furthermore relates to the optical glass fibre provided with a protective organic coating formed in accordance with a method wherein an N_2O -containing gas is used as said gas.

CLAIMS

1. A method of applying a protective organic coating to an optical glass fibre or a coated optical glass fibre, wherein said glass fibre is drawn from a preform and passed through a liquid which contains the material for forming said organic coating, once the amount of liquid coating material to be applied to the fibre has been adjusted, said coating material is hardened, while a gas is passed along the liquid, characterized in that nitrous oxide (an N_2O -containing gas) is used as said gas.
2. A method according to claim 1, characterized in that said nitrous oxide is introduced to said liquid at the upper side, at the place where the fibre is supplied.
3. A glass fibre provided with a protective organic coating, characterized in that said glass fibre has been obtained by using a method as defined in claims 1 - 2.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/NL 99/00383

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C03C25/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C03C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>JOCHEM C M G ET AL: "HIGH-SPEED BUBBLE-FREE COATING OF OPTICAL FIBRES ON A SHORT DRAWING TOWER"</p> <p>INTERNATIONAL CONFERENCE ON INTEGRATED OPTICS AND OPTICAL FIBRE COMMUNICATION (IOOC) AND EUROPEAN CONFERENCE ON OPTICAL COMMUNICATIONS (ECOC), VENICE, OCT. 1 - 4, 1985,</p> <p>vol. 1, no. CONF. 5, 11,</p> <p>1 October 1985 (1985-10-01), pages 515-518, XP002006367</p> <p>ISTITUTO INTERNAZIONALE DELLE COMUNICAZIONI</p> <p>cited in the application</p> <p>page 516, line 7 - line 20; table 2</p> <p>---</p> <p>-/--</p>	1-3

☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

17 September 1999

Date of mailing of the international search report

27/09/1999

Name and mailing address of the ISA

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Reedijk, A

INTERNATIONAL SEARCH REPORT

International Application No

PCT/NL 99/00383

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>EP 0 635 554 A (DOW CORNING CORPORATION) 25 January 1995 (1995-01-25) cited in the application column 3, line 22 -column 4, line 8 -----</p>	1-3

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/NL 99/00383

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 635554 A	25-01-1995	JP 7069685 A	14-03-1995

INTERNATIONAL SEARCH REPORT

onal Application No
PCT/NL 99/00383

A. CLASSIFICATION OF SUBJECT MATTER
IPC-6 C03C25/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C03C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>JOCHEM C M G ET AL: "HIGH-SPEED BUBBLE-FREE COATING OF OPTICAL FIBRES ON A SHORT DRAWING TOWER"</p> <p>INTERNATIONAL CONFERENCE ON INTEGRATED OPTICS AND OPTICAL FIBRE COMMUNICATION (IOOC) AND EUROPEAN CONFERENCE ON OPTICAL COMMUNICATI (ECOC), VENICE, OCT. 1 - 4, 1985,</p> <p>vol. 1, no. CONF. 5, 11,</p> <p>1 October 1985 (1985-10-01), pages 515-518, XP002006367</p> <p>ISTITUTO INTERNAZIONALE DELLE COMUNICAZIONI cited in the application</p> <p>page 516, line 7 - line 20; table 2</p> <p style="text-align: center;">---</p> <p style="text-align: center;">-/--</p>	1-3

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents:

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

17 September 1999

Date of mailing of the international search report

27/09/1999

Name and mailing address of the ISA
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Reedijk, A

INTERNATIONAL SEARCH REPORT

International Application No.

PCT/NL 99/00383

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>EP 0 635 554 A (DOW CORNING CORPORATION) 25 January 1995 (1995-01-25) cited in the application column 3, line 22 -column 4, line 8 -----</p>	1-3

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/NL 99/00383

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 635554 A	25-01-1995	JP 7069685 A	14-03-1995

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 40873/Vk/mb	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/NL99/00383	International filing date (day/month/year) 21/06/1999	Priority date (day/month/year) 26/06/1998
International Patent Classification (IPC) or national classification and IPC C03C25/02		
Applicant PLASMA OPTICAL FIBRE N.V. et al.		



1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 6 sheets, including this cover sheet.

☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

 These annexes consist of a total of 1 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☒ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand 29/10/1999	Date of completion of this report 20.09.2000
Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Fortunati, T Telephone No. +49 89 2399 8561 

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/NL99/00383

I. Basis of the report

1. This report has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.*):

Description, pages:

1,2 as originally filed

Claims, No.:

1-3 with telefax of 11/05/2000

2. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims 1,2
	No:	Claims 3
Inventive step (IS)	Yes:	Claims 1,2
	No:	Claims 3
Industrial applicability (IA)	Yes:	Claims 1-3
	No:	Claims

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/NL99/00383

2. Citations and explanations

see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:

see separate sheet

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/NL99/00383

- 1) Reference is made to the following documents:

D1: JOCHEM C M G ET AL: "HIGH-SPEED BUBBLE-FREE COATING OF OPTICAL FIBRES ON A SHORT DRAWING TOWER" INTERNATIONAL CONFERENCE ON INTEGRATED OPTICS AND OPTICAL FIBRE COMMUNICATION (IOOC) AND EUROPEAN CONFERENCE ON OPTICAL COMMUNICATIONS (ECOC), VENICE, OCT. 1 - 4, 1985, vol. 1, no. CONF. 5, 11, 1 October 1985 (1985-10-01), pages 515-518, XP002006367 ISTITUTO INTERNAZIONALE DELLE COMUNICAZIONI

D2: EP 0 635 554 A (DOW CORNING CORPORATION) 25 January 1995 (1995-01-25)

- 2) Regarding Section VII:

- 2.1) It is not evident where is the support in the originally filed disclosure of the following terms:

- a) " continuously " (see claim 1, line 5 of the claim page 1 submitted with Applicant's letter of 10.05.00)
- b) " body " (see claim 1, line 7 of the claim page 1 submitted with Applicant's letter of 10.05.00)
- c) " curing " (see claim 1, line 10 of the claim page 1 submitted with Applicant's letter of 10.05.00)

Moreover, the term " body " appears to be totally superfluous. The Applicant should simply indicate " ... through an organic liquid coating composition ... " instead of " ... through a body of an organic liquid coating composition ... ". The meaning of the two formulations is not different. As a consequence, it is not evident why one should insert in claim 1 the non-acceptable term " body " if the scope of protection is the same.

3) Regarding Section V:

3.1) Regarding process-claims 1 and 2:

Since the formulation of claim 1 is not acceptable for the reasons indicated in point 2) above, it is only possible to provide a preliminary analysis of the patentability of the claimed subject-matter as far as novelty and inventive step are concerned. This analysis is made in the assumption that the terms a) to c) above are either deleted or supported in the originally filed disclosure.

Regarding document D2, the use of nitrous oxide (a N₂O containing gas) in the present invention is totally different since said gas is used for displacing a coolant gas and air from the surface of the fiber prior to applying the liquid coating material, and not in the heating treatment as suggested by D2.

The inventors have noted that CO₂ as a purging gas created a lot of problems concerning the corrosion of the metal parts of the drawing apparatus. CO₂ reacts with moisture present in the ambient atmosphere and forms a corrosive compound. This compound affected adversely the strength of the fiber obtained. Therefore, the objective problem to be solved is to provide a purge gas which does not cause corrosion of the metal portions of the drawing apparatus and does not reduce the strength of the coated optical fiber. This problem is solved by using nitrous oxide (an N₂O containing gas) as said purge as indicated in claim 1 of the present application. The methods described in D1 and D2 are methods for the formation of protective coatings on optical fibers. None of the teachings of D1 and D2 would provide an incentive to use nitrous oxide (an N₂O containing gas) as a purge gas , as required in the present application. The documents D1 and D2 do not give any hint to the solution of the abovementioned technical problems since they are silent about nitrous oxide to be used as a purge gas. As a consequence, the claimed subject-matter is new and inventive over D1 and D2.

3.2) Claim 3 is a product claim. It refers to a glass fiber provided with a protective organic coating. However, a glass fiber provided with a protective organic coating is already known (see D1 and D2 and the cited prior art in the description of the application). The further feature of claim 3 (that is the fact that said glass fiber has been obtained by using a method as defined in claims 1 and 2) does not render the claimed glass fiber of claim 3 new and inventive over D1, D2 and the

prior art cited in the description of the application. A new process does not render a known product patentable. A product claim as product-claim 3 should be defined by means of product-features and not by means of process-features or of the process used to obtain it. Moreover, if a process is patentable, the product obtained by means of this process is automatically protected even if this product is not claimed. As a consequence, claim 3 should be deleted.

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Assistant Commissioner for Patents
United States Patent and Trademark
Office
Box PCT
Washington, D.C. 20231
ÉTATS-UNIS D'AMÉRIQUE

in its capacity as elected Office

Date of mailing:

06 January 2000 (06.01.00)

International application No.:

PCT/NL99/00383

Applicant's or agent's file reference:

40873/Vk/mb

International filing date:

21 June 1999 (21.06.99)

Priority date:

26 June 1998 (26.06.98)

Applicant:

BREULS, Antonius, Henricus, Elisabeth et al

1. The designated Office is hereby notified of its election made:



in the demand filed with the International preliminary Examining Authority on:

29 October 1999 (29.10.99)



in a notice effecting later election filed with the International Bureau on:

2. The election ☒ was



was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland

Facsimile No.: (41-22) 740.14.35

Authorized officer:

J. Zahra

Telephone No.: (41-22) 338.83.38

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference 40873/Vk/mb	FOR FURTHER ACTION see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. PCT/NL 99/ 00383	International filing date (day/month/year) 21/06/1999	(Earliest) Priority Date (day/month/year) 26/06/1998
Applicant PLASMA OPTICAL FIBRE N.V.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.



It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

- a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.



the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

- b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :



contained in the international application in written form.



filed together with the international application in computer readable form.



furnished subsequently to this Authority in written form.



furnished subsequently to this Authority in computer readable form.



the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.



the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. ☐ **Certain claims were found unsearchable** (See Box I).

3. ☐ **Unity of invention is lacking** (see Box II).

4. With regard to the **title**,



the text is approved as submitted by the applicant.



the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,



the text is approved as submitted by the applicant.



the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No.



as suggested by the applicant.



because the applicant failed to suggest a figure.



because this figure better characterizes the invention.



None of the figures.